Magnetic investigation of silver sheathed Sr$_{0.6}$K$_{0.4}$Fe$_2$As$_2$ superconductor

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Abstract

Magnetic investigation of a silver sheathed Sr$_{0.6}$K$_{0.4}$Fe$_2$As$_2$ tape prepared by ex-situ powder-in-tube technique (PIT) is reported. A transition temperature of 34.2 K was achieved. Dc magnetic measurements were performed in fields up to 14 T between 4.2 K and $T_c$. From hysteresis loops magnetic critical current densities $J_c$ were determined. The tape exhibits excellent $J_c$ performance. In low fields, the observed steep decline of $J_c$ in increasing field is comparable to that measured in MgB$_2$, although at a significantly lower absolute value. A kink-like crossover to a much flatter dependence at higher fields allows for a much better high field performance than that of MgB$_2$. Such kink is also visible in the field dependence of the mean activation energies $U$, which were determined from magnetic relaxation measurements. The obtained $U$ values are similar ($< 40$ meV at 4.2 K and 1 T) to those of Bi2212 tapes, but an order of magnitude smaller in comparison with good MgB$_2$ wires.

Keywords: Sr-122 tape, critical current density, magnetic relaxation, mean activation energy

1. Introduction

The discovery of superconductivity in MgB$_2$ in 2001 [1] aroused great interest in both scientific and technological community. Since then, this simple intermetallic compound with $T_c = 39$ K has been studied extensively [2, 3] and wires and tapes are already produced with industrial lengths. In 2008 a new family with $T_c$ in the same temperature range as MgB$_2$ was found, the iron-based superconductors LaFeAsO$_x$F$_y$ [4]. Shortly afterwards, the $T_c$ of these iron-pnictides was raised up to 55 K [5] by substituting La with alkali earth elements. Particularly, 122-type pnictides have large potential due to their relatively high $T_c$, huge $H_{c2}$ above 100 T [6, 7] and small anisotropy about 1.5-2 [8].
While displaying relatively low critical current density $J_c$ at low fields, 122-type pnictides have much higher $J_c$ at high fields and their dependence of $J_c$ on applied field is remarkably weak in comparison to MgB$_2$ [9, 10, 11].

The common preparation method of 122-type wires and tapes is based on powder-in-tube (PIT) process, because of its simplicity and low costs. However, there is a problem, because PIT-processed pnictides possess intrinsic weak links at grain boundaries and contain defects such as cracks, voids and impurities, thus causing the low $J_c$. These effects are unavoidable due to the mechanical deformation and heat treatment during the PIT process.

In order to prevent or reduce the described effects, various modifications to the preparation process of Ba$_{1-x}$K$_x$Fe$_2$As$_2$ (Ba-122) and Sr$_{1-x}$K$_x$Fe$_2$As$_2$ (Sr-122) are applied [11]. To limit void formation, an ex-situ route of the PIT process is used. Metal dopants are added to strengthen grain coupling and the superconductor is densified by optimum mechanical treatment.

2. Experimental

In this work we describe the study of a Ag-sheathed Sr$_{0.6}$K$_{0.4}$Fe$_2$As$_2$ tape with Sn addition produced by an ex-situ PIT process. A mixture of a nominal Sr:K:Fe:As = 0.6:0.5:2:2.05 ratio was ball-milled under Ar atmosphere, packed into a Nb tubes and annealed at 900° C for 35 h. The thus obtained Sr$_{0.6}$K$_{0.4}$Fe$_2$As$_2$ precursor powder was ground to powder in Ar atmosphere and mixed with 5 wt% Sn. Afterwards the powder was filled into a Ag tube with OD/ID of 8 mm/5 mm, respectively. After sealing the tube it was cold-worked by drawing and flat rolling to a tape with the superconductor cross-section area 0.1 x 4.3 mm$^2$. Finally, the tape was sintered at 850° C for 30 min.

Superconducting properties of the tape were investigated by magnetic measurements on a 5 mm long sample using the VSM option of a 14 T PPMS from Quantum Design. The field was applied parallel ($B||$) and perpendicular ($B\perp$) to the tape axis with the flat tape surface oriented parallel to the field (Figure 1). The used sheath material had no influence on the magnetic measurement. The transition temperature $T_c$, defined as the onset of diamagnetism and the transition width $\Delta T_c$, determined using a 10–90% criterion, were determined from zero field cooled measurements at 10 mT. In the temperature range 4.2–36 K (2 K steps) hysteresis loops between -2 T and +14 T with a constant field sweep of 6.3 mT/s were recorded. The extended Bean’s critical state model for orthorhombic samples [12, 13] was used to determine current densities $J_c$ assuming that the outer dimension of the superconducting core determines the current flow geometry.

![Figure 1 The sample is a cuboid. The direction of the current flow is indicated by arrows for each of the field orientations](image)

During hysteresis loop measurements the field sweep was interrupted in increasing and decreasing field to measure the change of magnetic moment up to 30 minutes. These magnetic
moment relaxations were measured at fields from 1 T to 13 T in 2 T steps. Using Anderson’s flux creep theory [14] the mean effective activation energy $U$ was determined.

3. Results and discussion

Figure 2 shows the zero field cooled curve measured at 10 mT. The superconducting transition is observed at 34.2 K and its width is 7.1 K. Compared to results for a Sr-122 tape presented by Lin [15], the value of $T_c$ is slightly lower (36.0 K and 34.7 K for sample 1 and 2 in [15], respectively) and the transition is sharper (9 K for sample 1 and 11 K for sample 2, estimated from figure 1 in [15]).

![Figure 2 Zero field cooled curve for the Sr-122 tape](image)

In Figure 3 hysteresis loops at three temperatures are shown. Measured magnetic relaxations are visible as spikes. Unlike MgB$_2$ superconductors, no flux jumps are present at low temperatures and low fields.

![Figure 3 Examples of hysteresis loops for B⊥ with measured magnetic moment relaxations at 4.2 K (outer curve), 10 K (middle curve) and 20 K (innermost curve)](image)
From the loops, the critical current density \( J_c \) was determined using the extended Bean´s critical state model [12, 13]. Measurements were made with applied field parallel and perpendicular to the tape axis to determine \( J_c \) anisotropy. Figure 4a shows \( J_c \) in both directions at selected temperatures. Higher \( J_c \) values are obtained when the field is applied perpendicular to the tape axis. The anisotropy (\( J_c \perp / J_c \parallel \) ratio) is quite small (from 1.1 at 3 T to 1.3 at 12 T), and it vanishes with increasing temperature. A somewhat stronger \( J_c \)-anisotropy was observed by Matsumoto et al. [16] in hot pressed Sr-122 tapes, where \( J_c \) as a function of external field directions to the tape surface was studied. The anisotropy values were almost two for all fields up to 3 T at 20 K and 30 K. Probably this anisotropy comes from a texturing during the preparation process. Such texture was e.g. reported in similar prepared Sr-122 tapes by Zhang et al. [11].

At 10 T and 4.2 K, \( J_c \) values reach \( 4.5 \times 10^4 \text{ Acm}^{-2} \) and at 14 T, \( J_c \) is above \( 3.0 \times 10^4 \text{ Acm}^{-2} \). Notably, up to 12 K, \( J_c \) values are still above \( 1.0 \times 10^4 \text{ Acm}^{-2} \) at 14 T. According to the field dependence of \( J_c \), it decreases very slowly with increasing field. This is well visible if compared with \( J_c \) of a good MgB\(_2\) wire (Figure 4b). Although \( J_c \) of MgB\(_2\) is much higher in low fields, it drops rapidly and at 9 T and 4.2 K it becomes lower than 1000 Acm\(^{-2}\). On the other hand, at 4.2 K and 10 K the Sr-122 sample exhibits very slow decrease of \( J_c \), which starts from about 3 T.

For the evaluation of \( J_c \) according to Bean´s critical state model it was assumed that the length scale for current flow is the sample dimension and that the material behaves in a non-granular way. Granularity could not be ruled out, especially because of the granular nature of the tape. E.g. Lin et al. [15] and Zhang et al. [11], showed that cold-worked or ordinary rolled tapes have a microstructure with many voids and residual cracks developed during the deformation process with the grain size around 2-5 \( \mu \text{m} \). Also the field dependence of \( J_c \) is strongly suggestive of granular behavior, as a steep decrease at low and a plateau-like behavior at high fields is typical of grain-boundary weak link driven current transport [17]. On the other hand a rough estimate of the characteristic length scale for current flow according to Angadi et al. [18] gives values which are in the order of the sample dimension, much larger than the size of the physical grains, thus supporting our assumption of non-granularity. In any case, using the sample dimensions for calculation gives a lower limit of the magnetic critical current density \( J_c \).

![Figure 4 Magnetic field dependence of critical current density \( J_c \) (a) and comparison (b) between Sr-122 and MgB\(_2\) [21]. The applied fields up to 14 T were parallel (dashed line) and perpendicular (solid line) to the tape axis](image-url)
Similar Sr-122 and Ba-122 tapes were studied in the works of Zhang [11], Lin [15] and Gao [19]. Lin produced tapes using ordinary and modified precursors prepared in a two-step sintering process and deformed them by conventional rolling and hot pressing. All tapes have a weaker field dependence than the presented tape, however, the conventionally rolled tapes have lower absolute values of $J_c$ and up to approximately 7 T $J_c$ of the presented tape surpasses that of the hot pressed tapes [15]. Best results so far were achieved for a hot pressed Sr-122 tape [20] with $J_c = 1.2 \times 10^5$ Acm$^{-2}$ at 10 T and 4.2 K and Ba-122 tape [19] with $J_c$ almost $10^5$ Acm$^{-2}$ under the same conditions.

In order to investigate the pinning behavior, magnetic relaxation measurements were performed on the tape. Within the measuring time interval the time dependence of the magnetic moment is in first approximation logarithmic (Figure 5), allowing to calculate the normalized creep rate $S = \frac{1}{M_{0,irr}} \frac{dM}{d\ln t}$, where $M_{0,irr}$ is the irreversible magnetization at the beginning of a relaxation. The irreversible magnetization was obtained by subtracting the reversible part from the measured magnetization. The reversible magnetization was determined as the middle of the hysteresis loop, assuming pure volume pinning.

![Figure 5 Typical time dependence of the irreversible magnetization observed at various fields at 10 K. The slope increases with field.](image)

According to the Anderson’s flux creep theory, the time dependence of the irreversible magnetization is given by $M(t,T) = M_0(T) \left[1 - \frac{k_B T}{U(T)} \ln \left(1 + \frac{t_b}{\tau} \right) \right]$, where $M_0(T)$ is the magnetization at $t_b = 60$ s and $\tau$ is the relaxation time assumed to be in the interval $10^{-12} < \tau < 10^{-6}$s. Together with the equation for $S$ one gets for the mean effective activation energy $U(T)$

$$\frac{1}{S} + 17.9 \leq \frac{U(T)}{k_B T} \leq \frac{1}{S} + 31.7.$$  

The temperature dependence of the activation energy is plotted in Figures 6a and 6b for both field directions. For $\mathbf{B} \parallel$ above 3 T and for $\mathbf{B} \perp$ above 5 T, the activation energies are practically field-independent within measuring accuracy, in good agreement with the $J_c$-results. Upon initial decrease of $J_c$ occurring up to 3 T, $J_c$ drops considerably slower.
Figure 6c shows the $U(T)$-dependence of a MgB$_2$ wire at 1 T, 3 T and 5 T [21]. At 1 T the pinning energies are one order of magnitude higher than those of the Sr-122 tape. At 3 T the energies of the MgB$_2$ sample are still slightly higher. In comparison with high temperature superconductors, the obtained $U$-values are similar to those of YBCO and Bi$_{2212}$ [22], below 40 meV at 4.2 K and 1 T.

![Figure 6](image_url)

Figure 6 Temperature dependence of mean effective activation energy at various fields applied parallel (a) and perpendicular (b) to the tape axis. In (c) the $U$-$T$ dependence is shown for MgB$_2$ at 1 T, 3 T and 5 T [15]

4. Conclusion

The investigated Ag-sheathed Sr$_{0.6}$K$_{0.4}$Fe$_2$As$_2$ tape exhibits excellent $J_c$ performance. In low fields, the observed steep decline of $J_c$ in increasing field is comparable to that measured in MgB$_2$, although at a significantly lower absolute value. A kink-like crossover to a much flatter dependence at higher fields allows for a much better high field performance than that of MgB$_2$. This kink is also present in the field dependence of the mean activation energy $U$ obtained from magnetic relaxation measurements. The values of these mean pinning energies are similar to those of HTS, but much smaller than found in good MgB$_2$ wires.

References

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